

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C08F 290/06, 290/14, C09D 183/10		A1	(11) International Publication Number: WO 99/11683 (43) International Publication Date: 11 March 1999 (11.03.99)
(21) International Application Number: PCT/US98/17558		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).	
(22) International Filing Date: 25 August 1998 (25.08.98)			
(30) Priority Data: 08/923,234 4 September 1997 (04.09.97) US			
(71) Applicant: MINNESOTA MINING AND MANUFACTURING COMPANY [US/US]; 3M Center, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).			
(72) Inventors: STEGINK, David, W.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). KUMAR, Ramesh, C.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). BAERTICH, Eric; P.O. Box 33427, Saint Paul, MN 55133-3427 (US).			
(74) Agents: PETERS, Carolyn, V. et al.; Minnesota Mining and Manufacturing Company, Office Intellectual Property Counsel, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).			
<p>(54) Title: LOW ADHESION BACKSIZE COATING COMPOSITIONS</p> <p>(57) Abstract</p> <p>A low adhesion backsize (LAB) coating composition comprising at least two components, wherein a first component is a silicone-containing acrylate polymer and a second component is (a) a silicone-containing polymer different than the first component, (b) an octadecyl acrylate containing polymer, or (c) a mixture thereof. Furthermore, when the second component is a silicone-containing polymer, the silicone-containing polymer contains silicone segments pendant to the polymer backbone.</p>			

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	MW	Malawi	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Mexico	US	United States of America
CA	Canada	IT	Italy	MX	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

-1-

Low Adhesion Backsize Coating Compositions

Technical Field

This invention relates to low adhesion backsize (LAB) coating
5 compositions and more particularly to blended release agents.

Background of the Invention

The purpose of a release agent is to reduce, control or moderate the force required to peel a pressure sensitive adhesive off an adherend that has been coated with a release agent. While the use of a release agent is useful to help reduce,
10 control or moderate the peel force when unrolling a roll of tape, or removing an adhesive-coated article from a sheet or pad of sheets, the peel force must co-exist with the necessity of ink receptivity. Ink receptivity is desirable so that the adhesive-coated article may be printed or written upon.

A variety of release agents are known to those skilled in the art that provide
15 control of the peel force of a pressure sensitive adhesive. One variety, for example, are acrylate-based polymer release agents. A second variety being polymers based on fluorine-containing monomers, and a third variety based solely on silicone-containing monomers. A typical example of the third variety would be crosslinked polydimethyl siloxane, herein referred to as PDMS. Each of these
20 various release agents provides a variety of properties over a spectrum of values. For example, a 100% PDMS release agent provides excellent release properties, that is, reduced peel force, but is generally considered to have little or no ink receptivity. At the other end of the spectrum are alkyl group containing (meth)acrylate based release agents that provide marginal release properties, but
25 exhibit excellent ink receptivity (that is, writability).

As a matter of general formulation principle, blends of two components usually have properties intermediate between those of the individual components alone. Extending this to the situation where one component is of higher quality than another, one would expect that a blend of two or more components would

-2-

have properties between the lowest quality and highest quality component, but still inferior to the properties of the best components.

Generally, release agents that display good performance against several measures at once are uncommon. Carrying this further; low viscosity, high solids, 5 high efficiency, writable, printable, age stable, broad spectrum release agents are very rare indeed. When blending release agents, it is highly unlikely that improvements will occur in many parameters, if any of the parameters at once.

Furthermore, release agents that exhibit writability are generally not compatible with a broad range of adhesives. Most typically, they are compatible 10 only with a narrow range of adhesive formulations, possibly only with one specific adhesive formulation.

Summary of the Invention

Briefly, in one aspect of the present invention, a low adhesion backsize (LAB) coating composition is provided comprising at least two components, 15 wherein a first component is a silicone-containing acrylate polymer and a second component is either a silicone-containing polymer different than the first component, or an octadecyl acrylate containing polymer, or a mixture thereof.

A further aspect of the present invention is that when the second component is a silicone-containing polymer, then the first silicone-containing polymer contains 20 silicone segments pendant to the polymer backbone.

Advantageously, the LAB coating compositions of this invention exhibit excellent and improved release properties when coated onto substrates. Improved release properties are quantified by laminating various adhesives to the LAB, and subsequently measuring the force required to peel the adhesive off the release 25 agent. This test is referred to as Adhesion to Backsize or (ATB) and is typically measured against a reference adhesive tape.

In another aspect of the present invention, an improved low adhesion backsize coating is obtainable according to the following steps:

- (a) providing a low adhesion backsize coating composition
30 comprising at least two components, wherein the first component is

-3-

a silicone containing acrylate polymer and a second component is a silicone-containing polymer different than the first component, or an octadecyl acrylate containing polymer or a mixture thereof,

- (b) coating the low adhesion backsize coating composition onto
5 at least one major surface of a substrate,
- (c) drying the coated substrate, wherein the drying process
includes heated metal drying rolls, steam treatment or radio
frequency drying.

Unexpectedly, the unique LAB coating compositions of this invention are
10 more efficient in that less LAB need be applied to obtain equivalent release levels
than any LAB component alone. Furthermore, at equivalent coat weights, the
blended LAB compositions will have better release levels than any LAB
component alone.

Additionally, excellent writability using hand-held writing instruments and
15 ink receptivity of printer inks and the like is obtained. In compositions known to
those skilled in the art, writability of blends were simply intermediate between the
two components. In a few cases, one property of a blend might have been better
than the corresponding property of an individual component, but this improvement
would have been obtained at the expense of another property. For example,
20 writability of a blend might be better than the writability of the worst of the blend
components alone, but the blend would also exhibit release values worse than the
best component alone. This is typically the tradeoff seen when trying to improve
the writability of a promising release agent.

The blended LAB coating compositions of the present invention
25 unexpectedly exhibit excellent release and excellent writability. In most cases,
writability of the blends was very close to that of the best of the components alone.
In some cases writability was better than that of either component.

Similarly, in compositions known to those skilled in the art, the
unacceptably high viscosity of an otherwise promising release agent might be
30 reduced by blending it with a low viscosity release agent. Generally speaking,
these compositions would reach the goal of reduced viscosity at the expense of

-4-

other beneficial properties. Most typically, a reduction in viscosity is associated by a reduction in percent solids, or by worsened release or worsened writability.

It is highly desirable for release agents to have excellent release properties along with excellent ink receptivity. It is even more desirable to obtain other

5 beneficial properties such as good handleability, low cost, low tendency to foam, low viscosity and good aging properties along with the first two features. Such release agents are not known in the art. In general, release agents that deliver writability are not capable of providing extremely low release levels, for example 15 grams/cm or less.

10 In addition to the improvement in release stated above, a further improvement of the compositions of this invention over previously known materials is that they possess reduced viscosity (compared to the most viscous component) without an associated reduction in percent solids. This is particularly useful because it allows one to coat the compositions at higher solids levels than 15 previously possible. Coating methods such as gravure coating favor low viscosity materials and previously the only means to obtain this was dilution. In addition, viscous materials tend to capture entrained air when mixed, and this leads to foam formation. Compositions with reduced viscosity materials are less likely to physically entrap air because the air bubbles escape more quickly, and thus they are 20 much less likely to cause foaming problems.

As used in this application:

“waterborne” means that the polymeric base of the release agent is dissolved, dispersed, suspended or similarly carried in a vehicle consisting primarily of water.

25 ***Description of the Preferred Embodiment(s)***

Briefly in one aspect of the present invention, a low adhesion backsize (LAB) coating composition is provided comprising at least two components, wherein a first component is a silicone-containing acrylate polymer and a second component is either a silicone-containing polymer different than the first 30 component, or an octadecyl acrylate containing polymer, or a mixture thereof.

-5-

A further aspect of the present invention is that when the second component is a silicone-containing polymer, the first silicone-containing polymer contains silicone segments pendant to the polymer backbone.

Advantageously, LAB coating compositions of the present invention 5 exhibit improved release properties as measured by adhesion to backsize (ATB) and as measured by ATB using with other challenging adhesives. Unexpectedly, the improvements demonstrated by the unique LAB coating compositions of this invention permit the use of less LAB to obtain any given release level.

Additionally, excellent writability using hand-held writing instruments and 10 ink receptivity of printer inks and the like is obtained.

In blended LAB compositions known to those skilled in the art, writability was simply intermediate between the different components. In a few cases, one property of the blend might have been better than that property of a single component. For example, writability of a blend was better than either component 15 alone. However, the present invention provides a release coating composition that exhibits improved properties with respect to writability and peel force. Theoretically, it is expected a blend would exhibit the writability of component A (poor release) and the release characteristic of component B (poor writability). However, when blended, the LAB coating compositions of the present invention 20 unexpectedly have excellent release and excellent writability.

Furthermore, blends also gave significantly reduced viscosity. This is particularly useful because it allows preparation of coating compositions at higher solids levels.

Representative component polymers for the blends of this invention 25 include:

(1) Representative of the class of octadecyl acrylate-containing polymers would be a tetrapolymer of octadecyl acrylate, methyl acrylate, acrylic acid and acrylonitrile.

	ODA	25-65%
30	Methyl Acrylate	0 to 40%
	Acrylic Acid	3-15%

-6-

Acrylonitrile 10-35%

(2) Representative of the class of silicone-containing acrylate polymers with pendant silicone segments is a silicone grafted tetrapolymer of methyl acrylate, acrylic acid, NVP and SiMac.

5 Methyl Acrylate 10-45%
 Acrylic Acid 2-15%
 NVP 20-50%
 SiMac 10-50%

(3) Representative of a second class of silicone containing acrylate polymers would be a tetrapolymer of methyl acrylate, methyl methacrylate, methacrylic acid and MFS.

10 Methyl Acrylate 10-45%
 Methyl Methacrylate 10-50%
 Methacrylic Acid 2-15%
15 MFS 10-50%

As used in this application; "ODA" means octadecyl acrylate and "NVP" means N-vinyl pyrrolidone, an ethylenically unsaturated cyclic amine. "SiMac" refers to a free-radically polymerizable methacrylate functional silicone macromonomer having a number average molecular weight in the range of 10,000.
20 "MFS" means mercapto-functional polydimethyl siloxane, having a number average molecular weight in the range of 8,000 and containing 4-5% mercaptan functionality on a molar basis.

Various MFS monomers and MFS analogs are commercially available from Shin Etsu (Japan), KF-2001 being one such suitable grade. SiMac and SiMac-analogs are commercially available from Shin Etsu and from 3M.

Blended LABs of the present invention can be used as release coatings for any article having a substrate and at least one layer of adhesive coated thereon. For example, LABs of the present invention can be used in the construction of repositionable notes, such as Post-it® Notes, flags, such as Post-it® Flags, roll 30 tape, such as Scotch™ Magic™ Tape. Other articles would include signage, pads of tape, and the like. The construction of such articles at the minimum comprise a

-7-

- release layer coated on one surface of a substrate, the substrate and at least one coating of an adhesive coated on the other surface of the substrate. Furthermore, the blended LABs of the present invention can be used as release coatings for articles, such as release liners and are useful in making indicia receptive release
5 liner material compatible with a wide variety of adhesives. Such an article would consist primarily of a substrate and a layer of low adhesion backsize coating thereon.

LABs of the present invention are described by referring to the following nonlimiting embodiments:

- 10 *Case 1* is for a blend of one ODA-containing acrylate polymer and one silicone acrylate co-polymer. The silicone-containing acrylate polymer can contain backbone silicone segments, although silicone segments pendant to the polymer backbone are preferred.

- Case 2* is for a blend of two different silicone acrylate co-polymers.
15 Preferably, at least one of the polymers contains silicone segments pendant to the polymer backbone.

- Case 3* is for a blend of two separate silicone acrylate copolymers and a third ODA containing acrylate polymer. Preferably, at least one of the polymers contains silicone segments pendant to the polymer backbone.
20 The blend ratios of the first component to the second component are in range of 1:99 to 99:1, wherein if the second component is a mixture itself, the mixture of the second component and the third component is in the range of 1:99 to 99:1.

- Preparation methods for these blends, and for the three constituent
25 polymers are similar. The following preparation methods are illustrative of a method for preparing the constituent polymers and the corresponding blends. Methods other than those described can be used provided they produce the copolymer of interest. Furthermore, the exact amounts are only indicative of a specific example, similar ratios can also be construed from these illustrative
30 preparation descriptions.

-8-

Generally, polymers of the type used in the blends of this invention are solution polymerized at approximately 25-40% solids in organic solvents, such as ethyl acetate using conventional free-radical methods. The solvent/polymer solution is then dispersed in water by neutralization of the acid moieties with an
5 organic or inorganic amine, following by removal of the organic solvent by vacuum stripping.

Coating Methods

Generally, all of the blends of this invention are of suitable viscosity for coating with a slot die, by direct gravure, or by roll coating. The preferred slot die
10 for applying these LAB blends has a 14.6 cm wide x 0.038 cm deep orifice slot through which the material is extruded onto a backing. The die has approximately a 0.16 cm smoothing land following the orifice. This coating die is suitable for application levels between 4 and 10 wet grams wet/sq meter. Depending on the dry coat weight desired, the solids level of the LAB blend will need to be adjusted
15 appropriately so as to coincide with a wet coat weight of at least 4 g/sq meter and no more than 10 g/sq meter.

Coating Weight

For release coatings, typically coat weights range between 0.05 and 1.72 dry
grams/sq meter. For the blends of this invention, coat weights ranging between
20 0.21 and 1.0 dry grams/sq meter are more preferred. Most preferred are coat weights ranging between 0.5-0.9 dry grams/sq meter. In the following examples, a variety of coat weights were often coated so that the effect of coat weight on release could be assessed.

The objects, features, and advantages of the present invention are further
25 illustrated by the following examples, but the particular materials and amounts thereof recited in these examples, as well as other conditions and details, should not be construed to unduly limit this invention. All chemical reagents, monomers comprising the polymers, and other materials are commercially available, for example from Aldrich Chemicals (Milwaukee, WI) and known to those skilled in
30 the art unless otherwise stated or apparent. In the examples, all parts and percentages are by weight, unless otherwise indicated. All molecular weights

-9-

reported are number average molecular weights in grams/mol. Viscosity is reported in centipoises, as measured using a Brookfield viscometer with spindle #3 at 60 rpm. The following test methods were used to evaluate and characterize the low adhesion backsize compositions produced in the examples. ATB-810 refers to
5 "Adhesion to Backsize" measurements made using Scotch™ Magic™ tape 810, commercially available from 3M.

Examples

Generally, polymers of the type used in the blends of this invention are solution polymerized at approximately 25-40% solids in organic solvents, such as
10 ethyl acetate using conventional free-radical methods. The solvent/polymer solution is then dispersed in water by neutralization of the acid moieties with an organic or inorganic amine, following by removal of the organic solvent by vacuum stripping.

Preparation of ODA-containing acrylate polymer (Polymer 1)

15 An amber-colored quart bottle was charged with 102 grams of octadecylacrylate (ODA) (50% solids in ethyl acetate), 11 grams of methyl acrylate (MA), 25 grams of acrylonitrile (AN), 13 grams of acrylic acid (AA), 0.5 grams of 2,2'-azobisisobutyronitrile (AIBN) and 182 grams of ethyl acetate. The resulting mixture was purged with nitrogen for 3 minutes at 1 L/min, after which the bottle
20 was sealed. The sealed bottle containing the solution was tumbled in a constant temperature bath for 48 hours at 65°C.

To obtain a waterborne solution of the above copolymer, 261 grams of deionized water, 3.1 grams of ammonium hydroxide, and 100 grams of the above-prepared copolymer were homogenized in a suitable container and then the ethyl acetate was stripped off under vacuum to obtain a 10% solids solution in water.
25

Preparation of silicone grafted (meth)acrylate copolymer (Polymer 2)

A 16 oz. amber glass reaction bottle was charged with 39.1 grams of methyl acrylate (MA), 30.4 grams of N-vinyl pyrrolidone (NVP), 43 grams of acrylic acid (AA), 26.1 grams of 10,000 MW silicone macromer (SiMac), 2.5 grams of 2,2'-azobisisobutyronitrile (AIBN) and 150 grams of methyl ethyl ketone (MEK). The
30

-10-

resulting mixture was purged with nitrogen for 2 minutes at 1 L/min, after which the bottle was sealed. The sealed bottle containing the clear solution was tumbled in a constant temperature bath for 24 hours at 55°C, resulting in a very viscous milky solution to which was added 35.7 grams of methanol to reduce solids from 5 40% to 35%.

To obtain a waterborne solution of the above copolymer, 315 grams of deionized water, 1.2 grams of 30% ammonium hydroxide, and 100 grams of the above-prepared copolymer were homogenized in a suitable container. The organic solvents were stripped off under vacuum to obtain a 10% solids solution in water.

10 ***Preparation of (meth)acrylate grafted silicone copolymer (Polymer 3)***

A 16 oz. amber glass reaction bottle was charged with 50 grams of methyl acrylate (MA), 20 grams of methyl methacrylate (MMA), 5 grams of methacrylic acid (MAA), 25 grams of mercaptofunctional silicone, 0.25 grams of 2,2'-azobisisobutyronitrile (AIBN) and 150 grams of methyl ethyl ketone (MEK).
15 The resulting mixture was purged with nitrogen for 3 minutes at 1 L/min, after which the bottle was sealed. The sealed bottle containing the solution was tumbled in a constant temperature bath for 48 hours at 65°C.

To obtain a waterborne solution of the above copolymer, 351 grams of deionized water, 1.38 grams of 30% ammonium hydroxide, and 100 grams of the 20 above-prepared copolymer were homogenized in a suitable container. The organic solvents were stripped off under vacuum to obtain a 10% solids solution in water.

Preparation of blends:

All blends are prepared in the same manner, except for the weights of the components. Three examples of the blending technique are given below, which 25 anyone skilled in the art could easily adapt to prepare any given blend. The order of addition is not important.

Case 1 Blend

Preparation of a Case 1 blend containing 20% Polymer 1 and 80% Polymer 2, with the percentages based on the solids content of the constituent components.
30 Into a clean gallon jar, 1600 grams of Polymer 2 at 10.0% solids was weighed out.

-11-

Polymer 2 is a gelatinous liquid with a viscosity of 3450 cps. While mixing gently with a glass stir rod, 400 grams of Polymer 1 at 10.0% solids was poured in. Polymer 1 was a thin liquid with a viscosity of 10 cps. After the two components had been mixed gently for a few minutes, the blend was thoroughly combined 5 using an air mixer turning at about 200 rpm. The result was 2000 grams of a uniform mixture at 10.0% solids, with a viscosity of 1320 cps. To verify homogeneity, a sample was spun in a ultracentrifuge at 10,000 rpm for 15 minutes. No phase separation was visible.

Case 2 Blend

10 Preparation of a Case 2 blend containing 50% Polymer 2, and 50% Polymer 3.

In the laboratory, 50 grams solution of Polymer 3 (13-15% solids) was taken in a 250 ml round bottom flask and to this was added 50 grams solution of Polymer 2 (9-10% solids).

15 The resulting homogeneous mixture was agitated at 200 rpm for 10-15 minutes at room temperature (22°C).

A milky viscous solution was obtained . Solids, pH and viscosity of the blend are 12.5%, 8.0 and 4600 cps respectively.

Case 3 Blend

20 Preparation of a Case 3 blend containing 50% Polymer 1, 25% Polymer 2 and 25% Polymer 3, with the percentages based on the solids content of the constituent components. Into a clean gallon jar, 571 grams of Polymer 2 at 10.0% solids was weighed out. Polymer 2 is a gelatinous liquid with a viscosity of 3450 cps. Into a second container, 1143 grams of Polymer 1 was weighed out. Polymer

25 1 was a thin liquid with a viscosity of 10 cps. Using a glass stir rod, 286 grams of Polymer 3 at 20% solids was combined with Polymer 1 briefly, then that mixture was combined with the 571 grams of Polymer 2 already in the first container.

Polymer 3 was a thin liquid with a viscosity of 55 cps. After the two components had been mixed gently for a few minutes, the blend was thoroughly combined 30 using an air mixer turning at about 200 rpm. The result was 2000 grams of a uniform mixture at 11.4% solids, with a viscosity of 450 cps. To verify

-12-

homogeneity, a sample was spun in a centrifuge at 10,000 rpm for 15 minutes. No phase separation was visible.

Coating Method 1

An 8% solids sample of a 20%/80% blend of Polymer 1 and Polymer 2
5 was prepared as stated earlier. This dispersion was pumped with a gear pump to a slot die (14.6 cm wide x 0.038 cm deep orifice, 0.16 cm smoothing land) and applied at a wet coat weight of 9.4 wet g/m² to a 15.2 cm wide web of 20 g/m² wood-free paper. This corresponds to a dry coat weight of 0.75 dry g/m².

From the following example, someone skilled in the art can roll coat any of
10 the LAB blends of this invention.

Coating Method 2

An 8% solids sample of a 20%/80% blend of Polymer 1 and Polymer 2 was prepared as stated earlier. This dispersion was pumped with a centrifugal pump to the pan of a 3-roll coater. Suitable roll coaters are commercially available from a wide variety of sources, such as a Tint-A-Web roll-coater, available from Norman X. Guttmann, Inc. (Iselin, NJ). In the roll coater used, the metering gap is not independently adjustable. The coat weight is thus more or less "fixed" and can only be determined by measuring the amount of LAB blend consumed during a given period of coating. In general, the 3-roll coater applied between 4.3 and 6.5
20 wet g/m² to a 15.2 cm wide web of 75 g/m² wood-free paper. It should be noted that the coat weight that results from this type of coater is affected partly by the viscosity of the blend and partly by the pan roll speed. Coating speed was 274 m/min. This corresponds to a dry coat weight of 0.34-0.52 dry g/m².

Drying Method 1

25 The web, having been previously coated with LAB using Coating Method 1 and remaining wet, is dried on two, 120°C, 17.8 cm diameter stainless steel heated cans at a speed of roughly 2.4 meters/minute, such that the final moisture content after drying is preferably in the range of 4-6% as measured gravimetrically.

-13-

Drying Method 2

The web, having been previously coated with LAB using Coating Method 1 and remaining wet, was dried using three forced air blowers. Each blower had a 15.2 cm x 2.5 cm orifice positioned perpendicularly to the web. Each blower 5 forced roughly 3-6 cubic meters/minute of 24°C air onto the web. The moisture content after drying was preferably in the range of 4-6% as gravimetrically measured.

Drying Method 3

The web was coated with LAB at a line speed 274 meters/minute using 10 Coating Method 2 and was dried using 4 ampere radio-frequency oven roughly 6 meters in total length. This oven was manufactured by Radio Frequency Corporation (Willis, MA). This rf oven has an airflow of 15-20 cubic meters/minute of 24°C air moving across the web. The moisture content after drying should preferably be in the range of 4-6% as measured gravimetrically.

15 *Preparation of Reference Tapes*

All "reference" tapes were generated by the following method. The adhesive of interest was coated at 6.7 dry grams/sq meter using an appropriate slot die or similar coating method at a speed of 15.2 meters/minute onto a web of 75 gram/sq meter wood-free paper approximately 95 micrometers thick. The adhesive 20 was coated from water. Two "reference" tapes were prepared (referred to hereinafter as "Reference Tape A" and "Reference Tape B":

Reference Tape A was made using the following adhesive:

97/1.5/1.0/0.5 IOA/HEMA/NVP/NaSS microspheres

The following suspension polymerization was run in a 2 liter glass resin 25 flask equipped with nitrogen inlet, low shear agitator, thermowell and condenser. To the flask the following is charged:

424.37 gms Distilled water

16.50 gms Stepanol AM-V (10% solution in water) (an alkyl sulfate commercially available from Stepan Co. Northfield, IL)

-14-

4.02 gms Triton QS-44 (potassium hydroxide neutralized pH=7; 41% solution) (commercially available from Rohm & Haas, Co., Philadelphia, PA)

9.35 gms Goodrite K-7600N (10% solution in water)

5 2.75 gms Sodium styrene sulfonate (NaSS)

These charges are mixed under a nitrogen purge with agitation at 200 rpm.

In a separate container the following charges are weighed:

533.50 gms IOA (isooctyl acrylate)

8.25 gms HEMA (hydroxyethyl methacrylate)

10 5.50 gms NVP (N-vinyl pyrrolidone)

0.11 gms t-DDM (tertiary-dodecyl mercaptan)

0.83 gms Luperox A75 (an organic peroxide commercially available from Elf Atochem North America, Philadelphia, PA)

15 0.19 gms Perkadox 16 (an organic peroxide commercially available from Akzo Nobel Chemicals B.V., The Netherlands)

These are mixed until the initiators are dissolved.

The agitation in the flask is increased to 360 rpm and the IOA mixture is added to the flask. Heat is applied via IR lamps to bring the contents to 42°C. It takes ten minutes to rise to this temperature. After approximately sixty minutes an 20 exotherm begins, rising to a peak temperature of 75°C in fifty-five minutes. The batch is cured at 75°C for six hours and the nitrogen purge is replaced with a nitrogen pad. After cooling the product is filtered through one layer of cheesecloth. The sample had a particle size from 35 to 45 micrometers.

To the resultant suspension the following components were added:

25 43.37 gms. Carbotac 26222 (51% solids in water) (an acrylic resin commercially available from B.F. Goodrich, Akron, OH)

1.15 gms. Kelzan S (a Xanthan gum commercially available from Monsanto Co., St. Louis, MO)

The batch is thoroughly mixed until uniform. Then enough ammonium 30 hydroxide is added to neutralize mixture to pH=7.5 -9.5. This mixture is referred to as "Adhesive A."

-15-

A reference tape made using Adhesive A was prepared as follows:

- Adhesive A was applied with a slot die onto 70 g/m² wood-free paper at 0.6 g/sq ft dry weight. Drying was accomplished on 120°C hot cans. This coat weight resulted in an adhesion level of roughly 47 g/cm as measured by 90 degree peel
- 5 adhesion to polyester film. Test results generated with this reference tape are referred to as "ATB-Adhesive A."

Reference Tape B was made using the following adhesive:

97/1.5/1.5 IOA/HEMA/NVP microspheres

- The following suspension polymerization was run in a 2 liter glass resin
- 10 flask equipped with nitrogen inlet, low shear agitator, thermowell and condenser.
- To the flask the following is charged:

587.00 gms Distilled water

35.00 gms Stepanol AM-V (10% solution in water)

35.00 gms Goodrite K-702 (ammonium neutralized pH=7; 10% solution in

15 water) (commercially available from B.F. Goodrich, Akron, OH)

5.25 gms HEMA (hydroxyethyl methacrylate)

5.25 gms NVP (N-vinyl pyrrolidone)

These charges are mixed under a nitrogen purge with agitation at 150 rpm.

In a separate container the following charges are weighed:

- 20 339.50 gms IOA (isoctyl acrylate)
- 0.07 gms t-DDM (tertiary-dodecyl mercaptan)
- 1.05 gms Perkadox 16

These are mixed until the Perkadox is dissolved.

- The agitation in the flask is increased to 340 rpm and the IOA mixture is
- 25 added to the flask. Heat is applied via IR lamps to bring the contents to 50°C. It takes fifteen minutes to rise to this temperature. After approximately ten minutes an exotherm begins, rising to a peak temperature of 76°C in eight minutes. The batch is allowed to cool back to 50°C and the nitrogen purge is replaced with a nitrogen pad. The flask is held at this temperature for a total heat time of 22 hours.
- 30 After cooling the product is filtered through one layer of cheesecloth. The sample had a particle size from 40 to 60 micrometers.

-16-

To the resultant suspension the following components were added:

27.67 gms. Carbotac 26222 (51% solids in water)

0.73 gms. Kelzan S

- The batch is thoroughly mixed until uniform. Then enough ammonium
 5 hydroxide is added to neutralize mixture to pH=7.5 -9.5. This mixture is referred
 to as "Adhesive B." A reference tape made using Adhesive B was prepared as
 follows:

Adhesive B was then applied with a slot die onto 70 g/m² wood-free paper
 at 0.6 g/sq ft dry weight. Drying was accomplished on 120°C hot cans. This coat
 10 weight resulted in an adhesion level of roughly 50 g/cm as measured by 90 degree
 peel adhesion to polyester film. Test results generated with this reference tape are
 referred to as ATB-Adhesive B.

Test Methods

	Properties	Test Methods
1	Viscosity	Brookfield viscosity
2	Percent solids	% Solids
3	Initial release performance	ATB-810, Adhesion to Backsize as measuring using 3M #810 tape.
4	Ink receptivity, as it pertains to hand held writing instruments.	Autopen Writability Test (AWT)
5	Printability, as it pertains to flexographic or lithographic printing	Densitomer reading of printed sample. (Ink Density)
6	Compatibility with a range of adhesives	ATB, as measured using "reference" tapes prepared with other adhesives ATB-Adhesive A ATB-Adhesive B
7	Age stability of release properties	ATB, measured after clamping in a hydraulic press and after accelerated aging.
8	Tendency to foam	Visual - observed level of foam

Description of Test Methods

- 15 **Brookfield Viscosity:** A liquid sample was placed in a small container, and measured using Brookfield spindle #3 at 60 rpm, except as noted specifically in the Examples. Brookfield viscometers can be obtained from Fisher Scientific.

-17-

Percent Solids: A small aluminum pan was desiccated at 120°C overnight and a tare weight was obtained. A small amount (1-5 grams) of the liquid to be measured was placed into the pan and the gross weight was determined. This pan was placed in a 120°C oven for one hour, after which the pan was cooled to room temperature and reweighed. The Percent Solids is calculated based on the dry weight, after subtracting the tare weight of the aluminum pan.

5 *ATB-810:* A 2.54 cm wide stripe of 3M #810 tape was laminated to the release surface using a 2 kg rubber roller, with two rolldown passes executed at about 20 cm/second. Within one minute after lamination, the adhesive stripe was
10 peeled off the surface at 30.5 cm/second using a 90 degree peel angle, and the force required to remove the strip at constant speed was measured. Suitable devices for measuring ATB-810 tape include a tensile testing machine such as one manufactured by MTS Systems Corp. (Eden Prairie, MN) or Instron, Corp. (Canton, MA)

15 *Autopen Writability Test:* This test is performed using an automatic writing device (such as one available from International Autopen, Sterling, VA), a variety of writing instruments, and an image analysis device. The reported result (AWT) is the amount of ink coverage (area percentage covered) in a 1 cm x 1 cm area. Generally speaking, at least 4 pens are used and the results are averaged together to
20 give a good indication of writability. Higher levels of ink coverage indicate better writability. Values are generally compared to a control surface such as office copy paper. In essence, an autopen controls pen angle, pen position and pen speed according to an analog template. An autopen delivers or "writes" ink in highly repeatable fashion much as a human hand deposits it during normal writing. A 60-degree pen angle is used. The test pattern is not particularly important, but it
25 should include straight lines, curved lines and changes in angle. One suitable pattern for use is an autopen "adjustment template" available from International Autopen. It is desired that the inked pattern should have sufficient line density such that between 15% and 35% of the area is covered by ink after writing. After
30 the ink is deposited, the sample is allowed to dry briefly. After drying, it is placed in an image analysis device that captures an image with a video microscope, then

-18-

digitizes a 1 cm x 1 cm area. Using the uninked area as a "white standard" and the darkest inked area as a "black standard," the number of dark pixels and light pixels are then counted, and the ratio is reported as an area percentage of dark pixels. The sample is lit circumferentially by a circular fluorescent tube. Suitable image analysis devices are available from Acuity Imaging Inc, of Nashua, New Hampshire.

ATB-Reference Adhesive: A 2.54 cm wide of a suitable "reference" tape is laminated to the release surface using a 2 kg rubber roller, with two rolldown passes executed at about 20 cm/second. Samples may be variably handled and tested after that point.

- i. If an initial value is desired, within one minute after lamination, the adhesive strip is peeled off the surface at 30.5 cm/second using a 90 degree peel angle, and the force required to remove the strip at constant speed is measured on a tensile testing machine. This result would be referred to as "ATB-Adhesive A," "ATB-Adhesive B" or "ATB-Adhesive #810".
- ii. If a more severe initial test of the release system is desired, the laminated sample is clamped in a hydraulic press at a pressure of 250 psi (176,000 kg/sq meter) for 30 seconds. Within 20 minutes of clamping, the adhesive strip is peeled off the surface at 30.5 cm/second using a 90 degree peel angle, and the force required to remove the strip at constant speed is measured on a tensile testing machine. Test results generated this way would be referred to as "Clamped ATB."
- iii. If a test of long-term stability of the release system is desired, the laminated sample (either after having been clamped or unclamped) is prepared, then put into an temperature and humidity controlled aging chamber for a period of time. Typically the sample would be aged at either 24°C and 50% RH, or 49°C and 5% RH for 3-14 days. After aging, the adhesive strip is peeled off the surface at 30.5 cm/second using a 90 degree peel angle, and the force required to remove the strip at constant speed is measured on a tensile testing machine. Test results

-19-

generated this way would be referred to as "Aged ATB" with a description of the aging conditions, and a description of whether the sample was clamped or not.

Example 1: ESCA data showing silicone functionality at the surface remains high despite reduced aggregate Si content.

ODA-containing polymers and silicone-containing polymers were blended according to Blend 1 preparation example above. The polymers were prepared according to the Polymer 1-3 preparation examples above using the component percentages set forth in Table 1. The coating compositions were coated onto paper at 0.86 g/m² dry and dried on 20 cm diameter 121°C hot cans. The coated papers were then evaluated on a Hewlett Packard 5950 ESCA system. The results are reported in Table 1.

Table 1

% Polymer (1) ODA Containing	% Polymer (2) Si Containing	Calculated %Si (Theoretical)	Atomic %Si (ESCA)	Excess %Si (Multiples)	ATB-810 (g/cm)
0	100	12.0	12.0	1.00	20.5
5	95	11.4	12.0	1.05	7.5
10	90	10.8	11.0	1.02	6.7
20	80	9.6	11.0	1.15	4.7
60	40	4.8	6.9	1.44	7.9
90	10	1.2	4.2	3.50	10.2
95	5	0.6	3.5	5.83	11.4
100	0	0.0	0.3	N/M	16.1

In Table 1, a theoretical calculation was prepared. That is, the value in the table is the % silicon that would have been expected in the LAB if the silicone were uniformly distributed throughout the LAB coating composition. For example, in the LAB compositions containing 80% of Polymer 2, of which 12% was silicon, the expected value would be $0.8 \times 12\% = 9.6\%$. A "multiple" was calculated for the percentage of extra silicon that was found at the surface. A value of 1.05 would mean that 5% more silicon was found at the surface than would be expected based on the actual content of silicon in the blend. Please note that a value of 5.83 would mean that almost 6 times as much silicon ends up at the surface than would be expected. While not intending to be bound by theory, the ESCA results illustrate a

-20-

phase separation in which silicone migrates to the surface. Note that the ATBs (adhesion to backsize) were lower for a 20/80 blend despite the slightly lower concentration of silicon at the surface.

Example 2: Viscosity data for blends of polymers (1) and (2)

5 The polymers were prepared according to the methods described earlier. Blends of polymers were prepared at different percentage ratios as set forth in Table 2. Final concentration of all samples was 10%. Viscosity was then measured using a Brookfield viscometer, spindle #3, 60 rpm. These blends were all coated at 0.75 g/m² dry coat weight and dried on 120°C hot cans. The viscosity
 10 and ATB of the blends are reported in Table 2. Note that very large reductions in viscosity were seen, along with large improvements in ATB-810.

Table 2

% Polymer (1) ODA Containing	% Polymer (2) Si Containing	Brookfield Viscosity (cps)	ATB-810 (g/cm)
100	0	10	16.1
95	5	13	11.4
90	10	19	10.2
85	15	26	10.6
80	20	33	11.8
60	40	125	7.9
40	60	435	6.4
20	80	1,320	4.7
0	100	3,450	20.5

Example 3:

15 A second set of samples were prepared using methods described earlier. In this example, three identical but separately compounded samples of one blend (20/80) and three separate obtained samples of a control (0/100) were prepared. These were coated on three separate occasions, and the samples were tested in double-blind fashion. In Table 3, "0/100" refers to Polymer 2 by itself, and
 20 "20/80" refers to a blend containing 20% Polymer 1 and 80% Polymer 2. Please note that in all three samples, better than a 50% improvement in ATB was noted. This indicates the improvement is repeatable.

-21-

Table 3

Replicate No.	ATB-810 (0/100 blend) g/cm	ATB-810 (20/80 blend) g/cm
1	20.5	8.7
2	21.7	9.8
3	21.3	9.0

Example 4:

Examples of a 20/80 blend of Polymer 1 and Polymer 2 were prepared at 5 10% solids. These were applied to 70 g/m² wood-free paper at 0.54 g/m² dry coat weight using a 3-roll coater at 274 m/min as described earlier. In this example, all variables were held constant except for drying method. Various drying methods were used as set forth in Table 4.

Table 4

Examples	Drying Method	ATB-810 (g/cm)
A	66°C air flotation	30.7
B	149°C air flotation	25.2
C	4 amp rf drying	15.0

10

Drying method affected release. In this example, one can see that increased heat resulted in improved release as measured by ATB-810 as reported in Table 4. Radio frequency (rf) drying seemed to give particularly good results. In rf drying, water molecules are excited quickly and volatilize with a burst of steam. It is felt 15 that this burst of steam is highly preferred in attaining the best release level possible.

Example 5:

A set of samples were coated of the same 20/80 blend as described in Example 3. As described in Example 4, a 3-roll coater was used to apply the LAB 20 at about 0.65 dry grams/sq meter. Line speed was approximately 457 meters/minute. Drying was accomplished in a three-zone air flotation oven followed by a heat treatment step on 120°C steel cans. Residence time on the steel cans was approximately 0.8 second. Coated samples were prepared with and

-22-

without the use of a steaming device. The steamer used was a "Coanda nozzle" full-web steamer of the type available from Thermo Electron, Inc. (Waltham, MA). The oven temperatures set forth in Table 5, were the average oven temperature for all three zones. Data in Table 5 is ATB as measured with #810 tape.

5

Table 5

ATB-810 (g/cm) of coated blend		
Oven Temperature	Steam ON	Steam OFF
93°C	8.7	22.8
135°C	9.8	13.8

The results reported in Table 5 show that the effect of steam dominated the effect of heat.

10 The data in Table 6 verifies that benefits of steam and temperature were also seen with a second adhesive system. Clamped ATB was measured using Reference Tape B coated at 6.2 dry grams/sq meter. As stated earlier, ATB was measured after clamping samples in a hydraulic press to create a more intimate bond between adhesive and release coating.

Table 6

Clamped ATB-Adhesive B of coated blend (g/cm)		
Oven Temperature	Steam ON	Steam OFF
93°C	8.1	15.7
135°C	5.9	9.2

15 **Example 6:**

Polymers 1 and 2 were prepared as described earlier. Different blend ratios, recorded in Table 7, of Polymer 1 and Polymer 2 were prepared at 10% solids. These were applied at 0.75 g/sq meter dry weight to 70 g/sq meter wood-free paper at 15.2 meters/minute using a slot die. Drying was accomplished on 120°C stainless steel hot cans. All other variables were held constant except blend ratio. The various blends were tested for initial release with #810 tape (commercially available from 3M) and also with a Reference Tape. To show the significant change after aging, ATB was measured with Reference Tape B after accelerated

-23-

aging at 24°C and 80% RH for 14 days. These three measures of release performance are reported in Table 7.

Table 7

% Polymer 1 ODA Containing	% Polymer 2 Si Containing	ATB-810 (g/cm)	ATB -Adhesive B (g/cm)	Aged 14 days (24°C/80% RH) ATB-Adhesive B (g/cm)
0	100	15.0	4.3	5.9
10	90	6.7	2.4	6.7
20	80	6.3	2.4	3.9
30	70	7.1	3.1	4.7
100	0	16.1	6.3	20.5

5 The data in the table shows that the 20/80 blend had better release than either component alone. This result was seen with # 810 tape and Adhesive B. The improved performance of the 20% and 80% blend was also seen after high humidity aging, indicating stable compatibility.

Example 7:

10 This example shows the effects of blending together two different silicone containing polymers. Polymer 2 is a silicone grafted (meth)acrylate copolymer with pendant silicone segments. Polymer 3 is a (meth)acrylate grafted silicone copolymer with backbone silicone segments. The various ratios are recorded in Table 8a and 8c. Samples listed in Table 8a were applied to 70 g/m² wood-free paper at 0.75 g/m² dry weight, and were dried at low temperature using 27°C forced air drying. Samples listed in Table 8c were applied using a roll coated followed by air drying at 24°C with coat weights ranging between 0.4-0.6 g/m². All samples were applied at the solids levels they were prepared at, although the wet coat weight was adjusted to maintain identical dry coat weights. No steaming
15 was used and no hot can treatment was used. Examples were tested for release using three different adhesives, (i) #810 tape, (ii) Reference Tape A and (iii) Reference Tape B. Reference Tape B was coated on sheet to sheet coater, such as described in U.S. Patent No. 5,487,780. The adhesion level of the Reference Tape
20

-24-

B was 17.3 g/cm to bond paper. ATB's with the various reference tapes were measured two ways, after clamping in a hydraulic press, and then after clamping plus 3 day aging at 49°C. Writability was measured by AWT as described earlier, wherein a higher % value indicates better writability. Writability was an average of 5 four pens and is recorded in Table 8a. ATB-810 results and viscosity data are recorded in Table 8c. ATB results with the reference tapes before and after aging are reported in Table 8b.

Table 8a

Sample	% Polymer (2) Si Containing	% Polymer (3) Si Containing	AWT Writability	% Solids of blend
D	0	100	14.9%	20%
E	20	80	17.3%	18%
F	50	50	18.6%	15%
G	80	20	19.5%	12%
H	100	0	19.8%	10%

Table 8b

Sample	Clamped ATB (Adhesive A, unaged) (g/cm)	Clamped ATB- Adhesive A aged 14 days @ 49°C (g/cm)	Clamped ATB- Adhesive B aged 14 days @ 49°C (g/cm)
D	28.7	37.0	54.7
E	25.6	28.7	46.1
F	26.4	37.0	39.3
G	30.3	33.5	30.7
H	30.3	34.6	36.6

10

Table 8c

Sample	% Polymer (2) Si Containing 10% solids	% Polymer (3) Si Containing 15% solids	ATB-810 (g/cm)	Viscosity of blend (cps)	% Solids of blend
I	0	100	15.7	100*	15%
J	25	75	4.3	600*	13.75%
K	50	50	4.7	4600**	12.5%
L	75	25	15.5	8600**	11.25%
M	100	0	24.4	10000**	10%

* Spindle #1 – speed 10 rpm

** Spindle #3 – 5 rpm

-25-

The results with #810 tape indicate a synergistic effect occurs when blending Polymer 2 at 10 % solids with Polymer 3 at 15 % solids (see Table 8c). Note also that the ATB's were improved for both Adhesive A and Adhesive B after heat aging, though the optimal blend ratio was not the same for the two adhesives

5 (Table 8b). Sample E was the optimal blend for Adhesive A and Sample G was the optimal blend for Adhesive B. Also note that the writability of all of the blends were much better than for Polymer 3 alone, indicating that release was improved without harming writability. The viscosity improvements of the blends over Polymer 2 alone are also noteworthy (Table 8c). It should be noted that Polymer 3

10 by itself foams terribly when agitated. Blends of the two polymers did not foam.

Example 8:

These samples demonstrated the effects of blending together a (meth)acrylate grafted silicone material (Polymer 3) and an ODA-containing material, Polymer 1. In this case, the silicone polymer contained silicone groups in

15 the backbone. The polymers were prepared according to the procedures described earlier. The ratios for the blends are reported in Table 9. The blends were applied to 70 g/m² wood-free paper at 0.75 g/m² dry weight, and were dried using 24°C forced air drying. No steaming or hot can treatment was used. The samples were tested for release using #810 tape and Reference Tape A. ATB with Reference

20 Tape A was measured after clamping in a hydraulic press.

Table 9

Sample	% Polymer (1) ODA Containing	% Polymer (3) Si Containing	ATB-810 (g/cm)	ATB-Adhesive A (Clamped) (g/cm)
N	0	100	13.9	28.7
O	20	80	13.0	29.9
P	50	50	15.4	26.0
Q	80	20	18.1	31.1
R	100	0	22.0	41.3

An advantage of these particular blends was a dramatically reduced tendency to foam, when blends were compared to Polymer 3 by itself. Polymer 3 is

-26-

prone to foaming, and reducing its foaming potential without harming release was just as unexpected as the unexpected improvement in release levels.

Example 9:

The polymers were prepared and blended according to the procedures
 5 described above. Various ratios of three-way blends are reported in Table 10a. The three way blends used two different silicone containing polymers, one being a silicone grafted (meth)acrylate polymer, and the second being a (meth)acrylate grafted silicone polymer. The third component was an ODA-containing polymer. Blend samples were applied to 70 g/sq meter wood-free paper at 0.07 g/sq ft dry
 10 weight, and were dried using 24°C forced air drying. No steaming or hot can treatment was used.

The blend samples were tested for release with the same reference tapes referred to in Example 7, Table 10b. ATB with the reference tapes were measured after clamping in a hydraulic press, and also after 3 day aging at 49°C and 5% RH.
 15 AWT writability is also reported in Table 10a.

Table 10a

Sample	% Polymer (1) ODA Containing	% Polymer (2) Si Containing	% Polymer (3) Si Containing	ATB-810 (g/cm)	AWT Writability
S	100	0	0	22.0	16.5%
T	80	10	10	17.7	16.8%
U	50	25	25	19.7	18.3%
V	20	40	40	19.3	18.7%
W	0	50	50	20.0	18.6%
X	0	0	100	13.8	16.6%
Y	0	100	0	28.0	20.9%

Table 10b

Sample	ATB-Adhesive A Clamped (g/cm)	Clamped ATB- Adhesive A (aged 3 days @ 49°C) (g/cm)	Clamped ATB- Adhesive B (aged 3 days @ 49°C) (g/cm)
S	41.3	41.7	53.1
T	28.0	33.9	49.6
U	29.1	31.9	33.9
V	31.5	33.1	46.1
W	27.2	37.0	40.0
X	28.7	37.0	54.7
Y	30.3	34.6	36.6

5

Note that Sample U was the best LAB for Aged ATB with both Adhesive A and B. Sample U was a 50/25/25 three-way blend of the three polymers. Note that the write-on of this blend was 18.3%, higher even than the baseline writability for Polymer 1, an LAB with outstanding ink receptivity. The three-way blend was best 10 for aging against these adhesives and still delivered outstanding writability.

Example 10:
 This example showed that regardless of coat weight, the "release" values for LAB blends are better than for either component alone. This example shows the same performance was not obtained simply by increasing the coat weight of 15 either component.

To that end, three LAB's were coated onto 70 g/m² wood-free paper at 0.75 g/m² dry coat weight. The coatings were dried on 120°C hot cans at a speed of 15.2 m/min. The three materials coated were: (i) 100% Polymer 2, (ii) 100% Polymer 1 and (iii) a 20/80 blend of Polymer 1 and Polymer 2. All three LAB's 20 were coated at a wide range of coat weights between 0.4 and 1.7 dry g/m². This range spans "very light" to "extremely heavy." ATB was measured to assess release

-28-

and the results are set forth in Table 11, with lower ATBs indicating improved release levels.

Table 11

Coat Wt (g/sq meter)	ATB-810 (g/cm)		
	100% Polymer 2 (Si containing)	100% Polymer 1 (ODA containing)	Blend
0.43	31.3	28.5	14.6
0.65	29.7	27.4	15.1
0.86	21.9	17.9	9.1
1.1	19.0	17.6	8.9
1.3	17.8	18.7	7.6
1.5	17.3	17.6	7.2
1.7	16.5	18.4	8.0

5 Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and principles of this invention, and it should be understood that this invention is not to be unduly limited to the illustrative embodiments set forth hereinabove. All publications and patents are incorporated herein by reference to the same extent as if each individual
10 publication or patent was specifically and individually indicated to be incorporated by reference.

-29-

What is Claimed:

1. A low adhesion backsize coating composition comprising at least two components, wherein a first component is a silicone-containing acrylate polymer and a second component is (a) a silicone-containing polymer different than the first component, (b) an octadecyl acrylate containing polymer, or (c) a mixture thereof.
5
2. The low adhesion backsize coating composition according to claim 1, wherein when the second component is a silicone-containing polymer, at least one of the silicone-containing polymers contains silicone segments pendant to the polymer backbone.
10
3. The low adhesion backsize coating composition according to claim 1, wherein the blend ratio of the first component to the second component are in range of 1:99 to 99:1, and wherein the mixture of the second component is in the range of 1:99 to 99:1.
15
4. The low adhesion backsize coating composition according to claim 1, wherein the octadecylacrylate-containing polymer is an octadecyl acrylate/methyl acrylate/acrylic acid/acrylonitrile copolymer.
20
5. The low adhesion backsize coating composition according to claim 1, wherein one of the silicone-containing polymers is methyl acrylate/acrylic acid/N-vinyl pyrrolidone/methacryloxypropyl polydimethyl siloxane polymer, and the other silicone-containing polymer is methyl acrylate/methyl methacrylate/methacrylic acid/mercapto-functional polydimethylsiloxane polymer.
25
6. The low adhesion backsize coating composition according to claim 1, wherein the low adhesion backsize coating composition is a blend of one octadecylacrylate-containing polymer and one silicone-containing polymer.
30

-30-

7. The low adhesion backsize coating composition according to claim 6, wherein the silicone-containing polymer contains backbone silicone segments.
- 5
8. The low adhesion backsize coating composition according to claim 6, wherein the silicone-containing polymer contains pendant silicone segments.
- 10 9. The low adhesion backsize coating composition according to claim 1, wherein the low adhesion backsize coating composition is a blend of two different silicone-containing polymers.
- 15 10. The low adhesion backsize coating composition according to claim 9, wherein at least one of the silicone-containing polymers contains silicone segments pendant to the polymer backbone.
11. The low adhesion backsize coating composition according to claim 1, wherein the low adhesion backsize coating composition is a blend of two different silicone-containing polymers and an octadecyl acrylate-containing polymer.
- 20
12. The low adhesion backsize coating composition according to claim 11, wherein at least one of the silicone-containing polymers contains silicone segments pendant to the polymer backbone.
- 25
13. A process for obtaining a low adhesion backsize coating comprising the steps:
 - (a) providing a low adhesion backsize coating composition comprising at least two components, wherein the first component is a silicone containing polymer and a second component is (i) a
- 30

-31-

- silicone-containing polymer different than the first component, (ii) an octadecyl acrylate containing polymer or (iii) mixtures thereof,
- (b) coating the low adhesion backsize coating composition onto at least one major surface of a substrate, and
- 5 (c) drying the coated substrate, wherein the drying process uses heated metal drying cans, steam treatment, or radio frequency drying.
14. A process for obtaining a low adhesion backsize coating comprising the steps:
- 10 (a) providing a low adhesion backsize coating composition comprising at least two components, wherein the first component is a silicone containing polymer and a second component is (i) a silicone-containing polymer different than the first component, (ii) an octadecyl acrylate containing polymer or (iii) mixtures thereof,
- 15 (b) coating the low adhesion backsize coating composition onto at least one major surface of a substrate, and
- (c) drying the coated substrate to a moisture content less than 8%.
15. An adhesive coated article comprising a substrate, at least one layer of adhesive coated on a portion of one surface of the substrate and at least one layer of low adhesion backsize coating coated on a portion of the other surface of the substrate, wherein the low adhesion backsize coating comprises at least two components, wherein a first component is a silicone-containing acrylate polymer and a second component is (a) a silicone-containing polymer different than the first component, (b) an octadecyl acrylate containing polymer, or (c) a mixture thereof..
- 20
- 25
- 30
16. The adhesive coated article according to claim 15 wherein the substrate is a polymeric film or paper.

-32-

17. A release liner consisting essentially of a substrate and a layer of low adhesion backsize coating thereon, wherein the low adhesion backsize coating comprises at least two components, wherein a first component is a silicone-containing acrylate polymer and a second component is (a) a silicone-containing polymer different than the first component, (b) an octadecyl acrylate containing polymer, or (c) a mixture thereof.
5

INTERNATIONAL SEARCH REPORT

Int'l Application No
PCT/US 98/17558

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C08F290/06 C08F290/14 C09D183/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C08F C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 262 087 A (TACHIBANA ET AL) 16 November 1993 see claim 1	1-3, 9, 10
X	WO 96 05962 A (AVERY DENNISON) 29 February 1996 see claims 1-3	1, 3, 5
X	EP 0 781 811 A (DOW CORNING TORAY) 2 July 1997 see claim 1	1-3
X	EP 0 379 146 A (MENICON LTD) 25 July 1990 see claim 1	1, 3, 5
A	EP 0 421 588 A (3M) 10 April 1991 see claims 5, 7, 9	1

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority, claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

Date of mailing of the international search report

7 December 1998

29/12/1998

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Lentz, J

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 98/17558

Patent document cited in search report		Publication date		Patent family member(s)		Publication date
US 5262087	A	16-11-1993	JP	5124931 A		21-05-1993
WO 9605962	A	29-02-1996	US	5616629 A		01-04-1997
			AU	3325595 A		14-03-1996
			BR	9509141 A		25-11-1997
			CN	1168652 A		24-12-1997
			DE	19581750 T		17-07-1997
			GB	2306492 A,B		07-05-1997
EP 781811	A	02-07-1997	JP	9183903 A		15-07-1997
			US	5789485 A		04-08-1998
EP 379146	A	25-07-1990	JP	2188717 A		24-07-1990
			JP	2716181 B		18-02-1998
			JP	2213820 A		24-08-1990
			JP	2716187 B		18-02-1998
			JP	3043711 A		25-02-1991
			AU	4779890 A		26-07-1990
			CA	2007852 A		17-07-1990
			DE	69019382 D		22-06-1995
			DE	69019382 T		28-03-1996
			US	4954586 A		04-09-1990
EP 421588	A	10-04-1991	US	5032460 A		16-07-1991
			DE	69028674 D		31-10-1996
			DE	69028674 T		17-04-1997
			JP	3088815 A		15-04-1991
			US	5202190 A		13-04-1993